Diffraction Notes

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POLE FIGURE DETERMINATION

Because of the anisotropy of individual crystals, the physical properties of most polycrystalline materials are dependent upon any preferred orientation or “texture” which may be developed in the component crystals. For example, the texture developed by rolling and annealing a silicon iron transformer sheet can have a major impact upon the magnetic properties, and thus the efficiency, of transformer cores made from the sheet material. The formability of steel sheet depends upon the elastic and plastic properties, which depend upon the texture developed during rolling and annealing. The mechanical and electrical properties of thin films depend upon the preferred orientation developed in the film during the deposition process.

The preferred orientation in rolled sheet, extrusions or films is best described and quantified by a pole figure. Pole figures are either stereographic or equal area plots of a specific (hkl) pole (or crystallographic direction) density for the individual crystals compromising the sample. Lambda Research has developed software for both data acquisition and reduction, which provides uniform resolution of data collection on an equal area spherical net. Most commercial pole figure software utilizes fixed equal angular increment data collection (usually 5 deg.), leading to high resolution at the center and lower resolution at the edges of the pole figure. Lambda’s software spaces the data points evenly throughout the entire pole figure. Further, the pole figure resolution can be carried to provide high resolution when required or lower resolution for high speed data collection and reduced cost.

Pole figures are obtained using a stepper motor-driven Schulz back-reflection texture goniometer. Defocusing corrections referenced to random powder samples allow data to be collected reliably to 80 deg. from the center of the pole figure. The results may be presented in either times-random or normalized format as a contour plot. Software has been developed for single pixel resolution contour plotting on a dot matrix printer which allows the results to be transmitted by fax or photocopied. An example of our new technology applied to aluminum foil specimens is cited below.

X-Ray Diffraction Pole Figure Determination for Aluminum Foil

A common texture developed in cold-rolled aluminum sheet reportedly consists of a combination of the {110} and {112} planes oriented parallel to the rolling plane, with the <-112> and <11-1> lattice directions parallel to the rolling direction, respectively.

An aluminum sheet sample was prepared by stacking ten layers of 0.001 in. thick commercial aluminum foil with the rolling directions maintained parallel for each sheet. Multiple layers were used because of the transparency of the foil to copper radiation. The rolling direction was chosen as the reference direction and the sample was oscillated + -0.5cm in a plane parallel to the sample surface in order to integrate the diffracted intensity over the maximum surface area and as many individual crystals as possible.

The diffracted intensities of the {111} and {200} peaks were measured using copper K-alpha radiation on an automated Bragg-Brentano focusing geometry horizontal x-ray diffractometer. The intensity data were collected for one second intervals at increments of 2-deg. on an equal area spherical net throughout the central 80 deg. of the pole figure.

The background intensity measured on both sides of the diffraction peak for each angle of tilt was interpolated to determine the background intensity component of the diffraction peak. After subtraction of the background, the corrected pole figure was constructed in spherical coordinates by linear interpolation.

The {111} and {200} pole figures are presented in Figures 1 and 2, respectively. The pole figures are displayed as log-scales times-random stereographic contour plots with uniform 2 deg. resolution. The outer limit of data collection is shown by the dashed circle at 80 deg. The times-random data conversion was obtained by comparison to a compacted aluminum powder randomly oriented reference sample in accordance with ASMT Specification E81-77. The results were also corrected for defocusing intensity losses, as a function of the angle of tilt, employing a fifth order Chebychev polynomial fitted to the normalized diffracted intensity from the random aluminum powder reference sample as suggested by Witt.(2) The Chebychev polynomial fit reduces the effects of propagation of random error in the defocusing correction in the region of low diffracted intensity near the perimeter of the pole figure.

The {110} and {200} pole figures for the aluminum foil are in good agreement with published results for cold rolled 996% reduction) copper sheet, also and FCC material.(1) A noticeable difference between the published results for copper and the aluminum foil results is evident in the {200} pole figure (Figure 2) Where a region of four to eight times-random pole density can be observed in the center of the plot. The texture {100} <001> is listed as an ideal orientation for recrystallization of aluminum after cold rolling.(1) Therefore, some degree of recrystallization has probably occurred during annealing of the foil after cold rolling, resulting in the more complex texture observed.

References:

(1) Chin, G.Y., “Metallographic Principles: Textured Structures,” in METALS HANDBOOK, Vol. 8, pp. 231-232, 1973.
(2) Witt, F., private communication.

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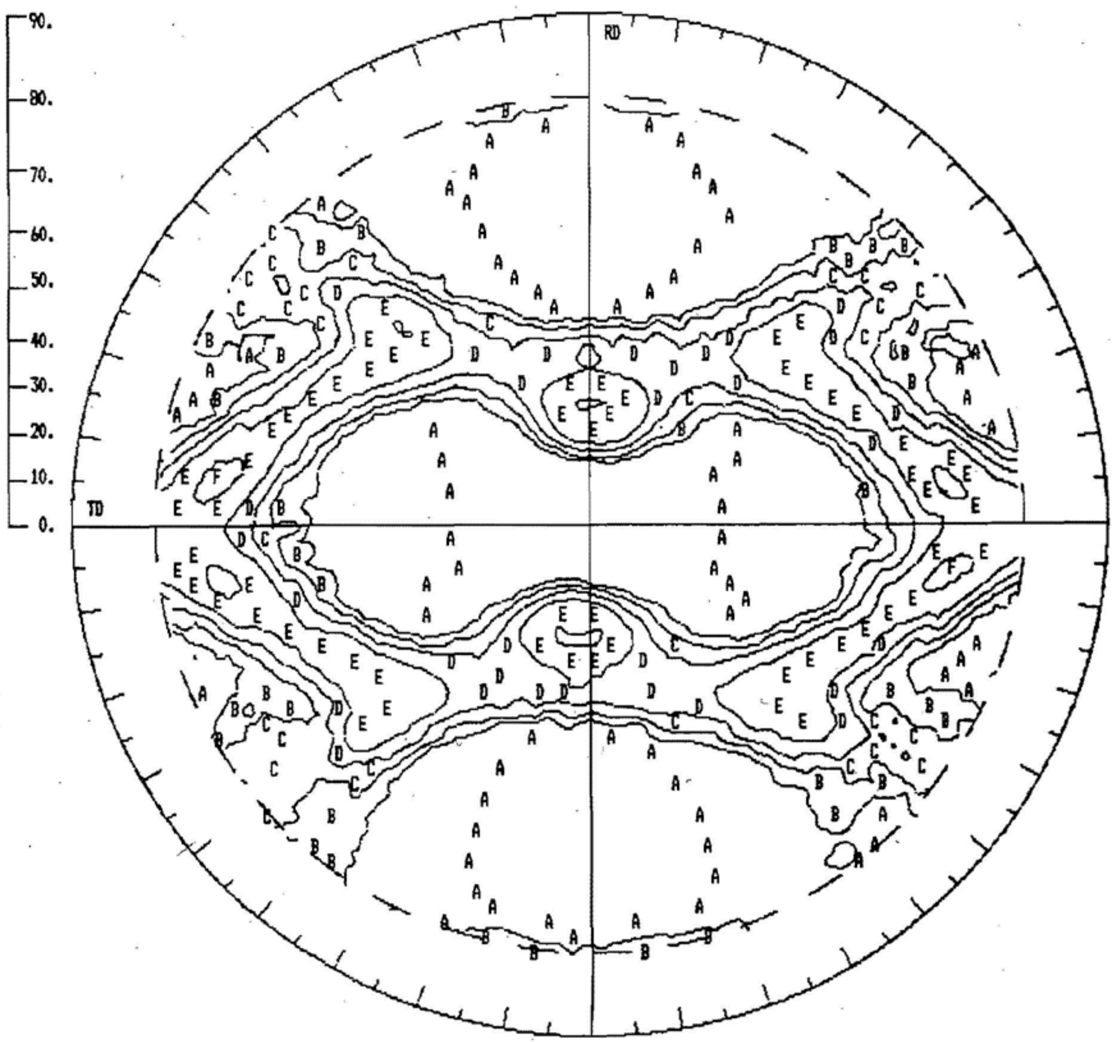
(111) Pole Figure
Stereographic Projection Plot, Times Random Format

Aluminum Foil Sheets 2/.7nspsh 1s



Sym.	x RANDOM
A	(.00- .24)
B	(.25- .49)
C	(.50- 1.00)
D	(1.01- 2.00)
E	(2.01- 4.00)
F	(4.01- 8.00)

2-Theta = 38.60 deg. Maximum intensity = 33700 Normal intensity = 1535
Lambda = 1.54178 Angstroms Background1 = 35.00 deg. Background2 = 41.00 deg.

Maximum Times Random = 4.73

Figure 1

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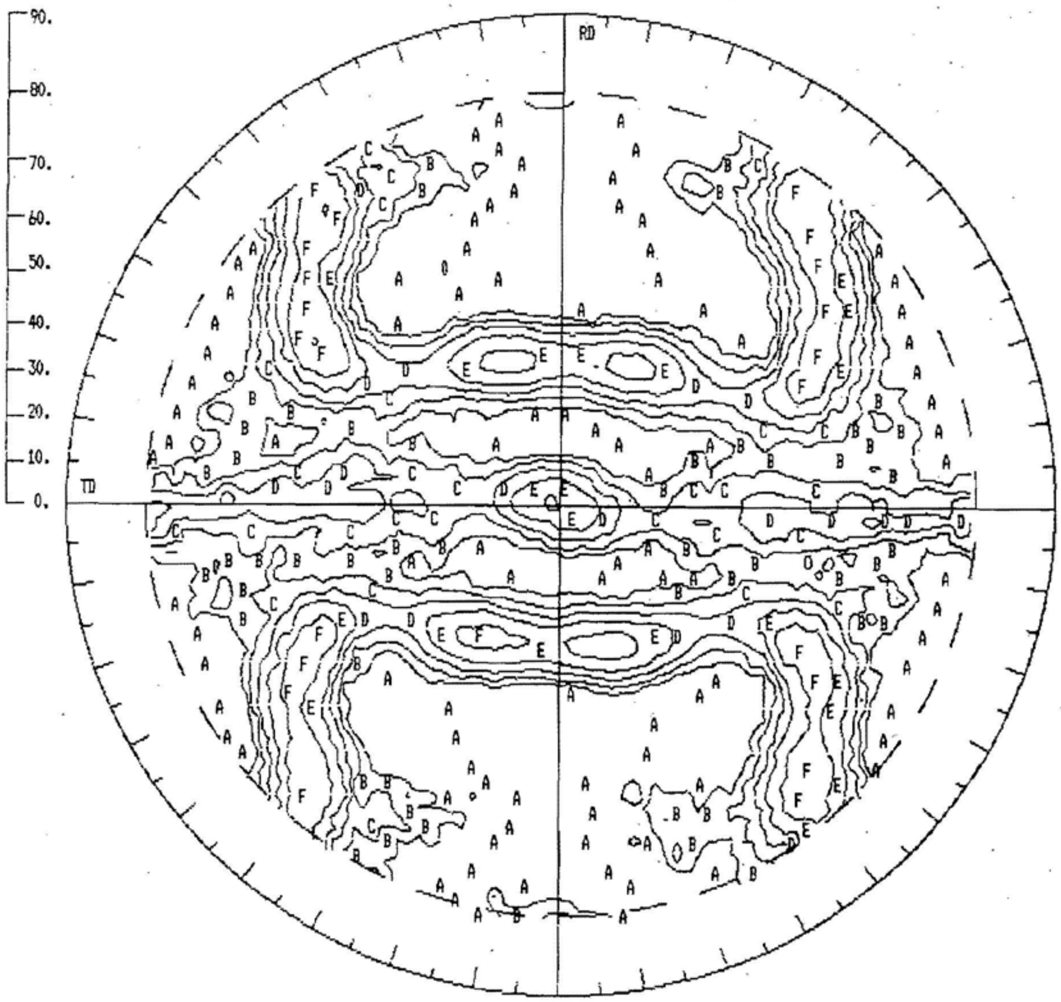
(200) Pole Figure
Stereographic Projection Plot, Times Random Format.

Aluminum Foil Sheets 2/.7nspsh 1s



Sym.	x RANDOM
A	(.00- .24)
B	(.25- .49)
C	(.50- 1.00)
D	(1.01- 2.00)
E	(2.01- 4.00)
F	(4.01- 8.00)
G	(8.01-16.00)

2-Theta = 44.80 deg. Maximum intensity = 33779 Normal intensity = 1100
Lambda = 1.54178 Angstroms Background1 = 42.00 deg. Background2 = 48.00 deg.

Maximum Times Random = 8.83

Figure 2

LABORATORY REMODELING

The entire main laboratory has been remodeled at Lambda Research. New metal laboratory cabinets and molded epoxy countertops have been installed throughout. Our new facilities provide specialized work stations for strain gaging, dimensional measurement, electropolishing, qualitative analysis, and pole figure sample preparation. Two 8 ft. hoods have been installed for electropolishing, ·and a 4 ft. hood for general wet chemistry. The new facilities will provide improved laboratory efficiency and better service to our clients.

TECHNOLOGY TRANSFER

Several of our long-standing clients have requested that the techniques, apparatus and software developed at Lambda Research for the measurement of residual stress and retained austenite be made available at their facilities. In order to meet our clients' needs, we have formed an affiliated corporation, Lambda Research Equipment Corporation, to provide apparatus and software under a technology transfer licensing agreement. This new service is available to those clients who require testing timeliness or quantities which could only be achieved on-site. The terms of the technology transfer agreement allow the use of our proprietary software, apparatus and sample preparation techniques by our clients' employees at their facility, and include apparatus maintenance, software updates and training. Our first installation has been in operation for six months, and a second system is under construction. For information on the details of the technology transfer agreement, contact Paul S. Prevey.

COMPUTER SYSTEM UPGRADE

The central computer system at Lambda Research is currently being upgraded to a 68020 microprocessor, with high speed cache memory and floating point processor support. The new system will provide general data reduction and office functions, and will contain the databases of our client and project history, the technical library, and complete archives of all data obtained at Lambda Research. The system will have two 190 megabyte hard disk drives, a 120 megabyte cartridge tape backup facility, and 6 megabytes of memory. The computer operates under a Unix-like operating system; running 24 hours a day for batch processing, and will be linked directly to the 68010 system running the seven diffractometers currently in operation in the laboratory. The new system will be four times faster in general applications than the 68000 system it replaces, and up to 100 times faster executing compiled Fortran code.